# Laboratory Environment Safety and Health Committee Cryogenic Safety Subcommittee

# MINUTES OF MEETING 03-04 July 28, 2003

## **FINAL**

Committee Members Present

M. Gaffney

R. Gill

M. Iarocci

S. Kane

P. Kroon

E. Lessard (Chairperson)

M. Rehak

R. Travis\* (Secretary)

K. C. Wu

(\* non-voting)

**Visitors** 

S. Hodgetts

M. Hucker

C. Petrovic

S. Shapiro

Agenda:

1. Review of Physics Department Magnetic Properties Measurement System

Minutes of Meeting: Appended on pages 2 through 4.

ESH COMMITTEE MINUTES APPROVED:

DM2120.

**Committee Members Absent** 

E. Lessard J & LESHC Chairperson

W. Glenn

P. Mortazavi

J. Muratore

J. Peters

Chairperson E. Lessard called the fourth meeting in 2003 of the Laboratory Environmental Safety and Health Committee (LESHC) to order on July 28, 2003 at 2:10 p.m.

- Review of Physics Department Magnetic Properties Measurement System
  (MPMS): E. Lessard invited C. Petrovic to present the proposed installation of a
  Magnetic Properties Measurement System in Room 2-103 of Building 510. Mr.
  Petrovic's presentation is included as Appendix 1 to these minutes. The preliminary
  (unreviewed) Physics Department Experimental Safety Review Form for this MPMS
  was reviewed by the Subcommittee in preparation for this meeting. (It is attached as
  Appendix 2.)
  - 1.1. Mr. Petrovic and other attendees made the following points during the course of the presentation and in response to specific Committee questions:
    - 1.1.1. This Magnetic Properties Measurement System (Quantum Design MPMS 7T) is a commercially available unit. The BNL Materials Science Department has a similar MPMS.
    - 1.1.2. The research group purchased the optional environmental shield, which reduces magnetic field strength by about 50% when compared to an unshielded model.
    - 1.1.3. The stored energy in the magnet is approximately 3.5 kilojoules, based on an inductance of 7 8 henrys and a current of 32 –33 amperes. The machine has quench protection circuit that is designed by the manufacturer.
    - 1.1.4. The machine stores 35 liters of liquid helium in the MPMS Dewar. Refilling is required every 3 to 7 days, depending on the use.
    - 1.1.5. A 100 liter storage dewar will be used for the helium refilling. This dewar will not be stored in the MPMS room, but it will be moved adjacent to the machine for refilling.
    - 1.1.6. The relief valves for the helium dewar are set at 1.5 psi.
    - 1.1.7. The liquid helium relief valves discharge through the venting path. The committee noted that it is standard BNL practice to have separate venting and pressure relief discharges.
    - 1.1.8. The machine has a liquid nitrogen blanket with a 40 liter capacity. The LN2 space has two relief valves set at 1.5 and 10 psi respectively as well as O-ring plugs for pressure relief.
    - 1.1.9. The Oxygen Deficiency Hazard (ODH) calculation, as presented to the Committee, assumed the complete volume of the MPMS helium dewar was evaporated. The refilling operation was not considered. The area that will house the storage dewar was not evaluated for ODH concerns.
    - 1.1.10. MPMS system trainees will be accompanied by trained personnel.
    - 1.1.11. The MPMS room is locked when unattended.
  - 1.2. The following motions were crafted by the Committee:
    - 1.2.1. Motion No. 1 Prior to performing MPMS commissioning activities, the Principal Investigator must:

- 1.2.1.1. Refer to the "Static Magnetic Field" Subject Area at <a href="https://sbms.bnl.gov/standard/1u/1u00t011.htm">https://sbms.bnl.gov/standard/1u/1u00t011.htm</a> and conform to all requirements Complete<sup>1</sup>.
- 1.2.1.2. Ensure that all personnel that are involved in the commissioning process (including the Quantum Design representative) take the BNL "Cryogen Safety", "Oxygen Deficiency Hazard" and "Static Magnet Field" training courses at <a href="http://training.bnl.gov/">http://training.bnl.gov/</a> Complete<sup>1</sup>.
- 1.2.1.3. Review the SBMS Subject Area "Oxygen Deficiency Hazards (ODH), System Classification and Controls"

  <a href="https://sbms.bnl.gov/standard/16/1600t011.htm">https://sbms.bnl.gov/standard/16/1600t011.htm</a>, perform the ODH calculations, and implement the required control measures for both the MPMS room and the liquid cryogen storage area. The postulated quantity of cryogen that is available for release must consider all MPMS operations, including helium transfers. Submit the required calculations and proposed control measures to the LESHC Cryogenic Subcommittee for review. (Complete, see Section 3, below for additional information.)
- 1.2.1.4. As required, install MPMS relief valve piping, ductwork, or deflectors to redirect the discharge of cryogens away from personnel Complete<sup>1</sup>.
- 1.2.1.5. Submit the MPMS commissioning work planning documentation to the Subcommittee for review Complete<sup>1</sup>.
- 1.2.2. Motion No. 2 At the end of the commissioning process, but prior to the start of MPMS operations, the Principal Investigator must:
  - 1.2.2.1. Implement Conditions 1.2.1.1, 1.2.1.2, 1.2.1.3 and 1.2.1.4, as appropriate for MPMS operation. Complete<sup>1</sup> except for the ODH issue. See Section 3 below for additional information.
  - 1.2.2.2. Provide the liquid helium dewar relief valve orifice area calculation, based on the maximum credible energy deposition rate in the helium volume **Complete**<sup>1</sup>.
  - 1.2.2.3. Reformat Attachments 2-6 of the MPMS Experimental Safety Review Form into a procedure. (The ESR is included as Appendix 2 to these Minutes.) Complete<sup>1</sup>
  - 1.2.2.4. Submit a Piping and Instrument Drawing (P&ID) to the Committee Complete<sup>1</sup>.
- 1.2.3. S. Kane made a recommendation for approval of the Motions.
- 1.2.4. Seconded by M. Iarocci.
- 1.2.5. The motions were approved by vote of 8 in favor and none opposed.
- 2. The Meeting was adjourned at 3:55 p.m.
- 3. Addendum to the Minutes
  - 3.1. The purpose of this addendum is to document the salient points of the Committee discussions, emails and site inspections that occurred after the 7/28/03 meeting.

<sup>&</sup>lt;sup>1</sup> This MPMC Commissioning or Operational prerequisite was completed during the review cycle for these minutes.

- 3.1.1. The ODH calculations were revisited in response to Condition 1.2.1.3 above. Assuming no ventilation, the MPMS room is ODH Class 1 when the 100 liter liquid helium dewar is present.
- 3.1.2. The 100 liter liquid helium dewar is present in the room only during the initial MPMS fill and subsequent refilling operations. It is stored in the large corridor outside the room.
- 3.1.3. For MPMS commissioning, the Committee agreed to allow the initial filling of the machine predicated on the short duration using the dewar, the availability of the extra volume in the corridor, and the use of a high velocity fan to assure active ventilation in the room.
- 3.1.4. One additional refill (scheduled for ~8/8/03) using the high velocity fan was permitted by the Committee.
- 3.1.5. Subsequent liquid helium fills require:
  - 3.1.5.1. Testing of the temporary ventilation configuration to assure that the high velocity fan is providing adequate air flow/exchange in the vicinity of the MPMS, or
  - 3.1.5.2. Completion and confirmatory testing of the permanent ventilation supply and exhaust for the MPMS room.
  - 3.1.5.3. The ODH calculations must be revised based on the test results of the selected ventilation configuration.

# Magnetic Fields

 Magnet Size
 1 Gauss, Radial
 5 Gauss, Radial
 1 Gauss, Axial
 5 Gauss, Axial

 MPMS 7T
 4S In(114.3 cm)
 20 In. (50.8 cm)
 20.5 In.(52 cm)
 2 In. (5.1 cm)

With environmental shield distances are ~ 50% smaller

Effect	Field (G)	Distance from Dewar (cm)
Effects on electron microscope	1	216
Disturbance of color computer monitors	1-3	152
Disturbance of monochrome computer monitors	3-5	127
Erasure of credit cards, bank cards, etc.	10	102
Effects on watches and micromechanical devices	s 10	102
Lowest known field affecting a pacemaker	17	8.4
Erasure of magnetic tapes	20	79
Saturation of transformers and amplifiers	5	58
Erasure of floppy disks	35	030

# Safety

- Never disconnect a charged magnet from the controllers
- Magnetic field around the MPMS should be measured and a line drawn to denote the region outside which the magnetic field does not exceed 5G
- Anyone wearing a pacemaker or similar device should stay at least 3.0-4.5 meters away from the MPMS dewar

There is no need to be close to MPMS when the magnet is on

### Cryogenic Aspects

Top of Level Meter

Top of Magnet

Magnet and pick up coil

center

Bottom of magnet

Flow impedance tube

100 %

50 %

40%

30%

### Cryogen sources:

- Liquid Nitrogen jacket 401
- Liquid Helium dewar 56i (vibration isolated)
- Storage Dewars
- Must be refilled every 4-7 days

Both the Temperature Control Module and helium dewar provided by Quantum Design employ relief vaives and burst disks to provide safe operation if there is either a leak into any of the insulating vacuum spaces or the superconducting magnet quenches

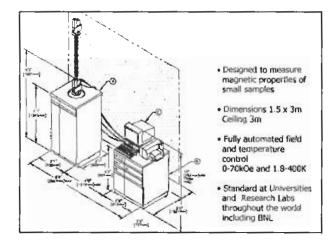
Protection during cryogen transfer: thermal gloves, eye protection, covered shoes

### Oxygen Deficiency

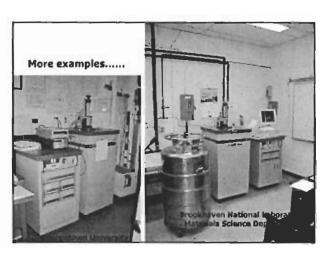
- . Density of He liquid 150g/I (Handbook Chem/Physics)
- Density of He gas 0.178g/l conversion factor 842.7
- 35 liters would become 29494dm<sup>3</sup> ~ 30m<sup>3</sup> of gas
- Room volume ~ 90m³, therefore oxygen content in the total room volume ~ 0.14
- Oxygen partial Pressure 106.4 Torr, estimated fatality factor F=0.0001
- Equipment failure rates: Dewar leak or rupture: (highest): 10\*/hr
  Fluid line leak: 5x10\*/
  Vacuum valve failure 1x10\*/day, 4.17x10\*/hr
  Weld leaft 5x10\*/day, 4.17x10\*/hr
  Manual valve plug 1x10\*/day, 4.17x10\*/hr
  Relief valve failure 1x10\*/day, 4.17x10\*/hr
  Total ~ 6.9x10\*
- Risk Factor (ODH fatality rate) is 7x10-10
- ODH class 0. requires posting and training.

# Magnetic Properties Measurement System

- Introduction
- Static Magnetic Fields
- Cryogenic Aspects
- Oxygen Deficiency







### **APPENDIX 2 TO LESHC 03-04**

Before using a copy of this form, verify that it is the most current version by checking with your Experiment Review Coordinator. Double-click to change the state of a checkbox.

### **EXPERIMENT SAFETY REVIEW FORM**

REVIEW NUMBER (supplied by ERC):				
PRINCIPAL INVESTIGATOR: Ced	omir Petrovic	DATE: July 16 <sup>th</sup> 2003		
GROUP:				
EXT:	E-MAIL: petrovic@bnl.gov	LIFE NUMBER: 22904		
Project Title: Measurement of mag	netic and transport properties of mater	rials		
Location(s): Building 510B, room	2-103			
Funding Source/Account Number				
Proposed Start Date and Duration	n: August 1 <sup>st</sup> 2003			
SIGNATURES:				
Principal Investigator:	Cedomir Petrovic	Date: July 17, 2003		
Experiment Review Coordinator:		Date:		
ESRC Chairman:		Date:		
		Date:		
Approval Department Chairperson:		Date:		
Review/Approval Comments:				
Walkthrough Signature:		Date:		
Expiration Date (max 1 yr.):				
FUA Change Required? ☐ Y ☐		anges Required? ☐ Y ☐ N		
Has a NEPA Review been Perforn	ned for this Project?			
Project Termination Acceptance	Signature:	Date:		
Comments:				

### I. DEFINE THE SCOPE OF WORK

### A. Description

Describe the experiment purpose/scope. Identify all apparatus that will be used, and associated requirements. List special equipment (X-ray generators, lasers etc.) that will be used during the project. Identify measurement and test equipment, apparatus operating conditions, and required maintenance procedures as appropriate. Include calibration frequency for formal <u>calibration requirements</u>. Attach supporting documents such as engineering calculations, drawings and specifications.

Indicate if modification of facility is required. Include the setup and decommissioning phases of the experiment. The Work Permit Process/Form may better address the hazards & controls of the set-up and/or tear down phases. Indicate if a Work Permit will be used.

The purpose of this experiment is to perform basic physical characterization of materials by measuring magnetic and electrical transport properties. Magnetic properties measurement system (MPMS) will be used. See Attachment 3: System description.

#### B. Materials Used /Waste Generated

List materials to be used and wastes generated. Refer to the <u>BNL Chemical Management System</u> for a complete listing of the chemicals in your locations. Include samples, chemicals, controlled substances, gases, cryogens, radioactive materials, and biological material. You may use generic chemical class descriptions for commonly used materials (e.g., organic solvents, acids). List disposal methods. **Denote disposal method using the codes below.** 

Disposal Method Type		Estimated Annual	
(Code below)	Per Use	Total/Yr	Waste Generation
Т			
	30 liters	2000 liters	
	40 liters	2000 liters	
	Method Type (Code below)	Method Type (Code below)  T  (provide to per Use)  T  30 liters	Method Type (Code below)  Per Use Total/Yr  T  30 liters 2000 liters  40 liters 2000

Note: Identify Age Sensitive materials or special handling requirements.

### **Disposal Method Codes:**

Air Emissions	Liquid Effluents	Wastes
<b>P</b> = Point Source	S = Sanitary	H = Hazardous
<b>F</b> = Fugitive	ST = Storm water	I = Industrial (Non-hazardous waste e.g., oils)
	O = Other	R = Radioactive
		M = Mixed (Radioactive + Hazardous)
		RM = Radioactive Medical
		MW = Medical
		T = Trash

### C. Waste Minimization/Pollution Prevention

Describe how you plan to minimize generation of the wastes described above, and identify pollution prevention opportunities. Consider Ordering/using the smallest amount, using recycled material substituting non-hazardous materials. The <u>Pollution Prevention and Waste Minimization Subject Area</u> describes how to plan, conduct, and closeout work activities to eliminate or minimize the impact of their activities on the environment.

There will be no waste generated.

### II. IDENTIFY AND ANALYZE HAZARDS ASSOCIATED WITH THE WORK

In this section indicate the hazards in each class. Include the setup and decommissioning phases of the experiment.

Physical Hazards (check all that apply) ☐ None						
✓ Cryogens	✓ Oxygen deficient atmosphere □ Noise > 85 dBA			oise > 85 dBA		
☐ Fall hazards (e.g., ladde	ers, elevated pla	tforms, tow	ers)			
☐ Material handling equip	ment (e.g., crane	es, hoists, f	orklift	s)		
☐ Machine shop or nonpo	rtable powered t	ools use				
☐ Electrical hazards (expo	sed conductors	, large batte	eries,	capacitors, etc)		
☐ Confined space			□ Tr	enching/soil excavation	n	
☐ Extreme temperatures			□R€	emote location		
☐ Other (specify):						
Pressure or Vacuum Sys	stems (check all	that apply)		□ None		
☑ Compressed gases (led	ture bottles, cyli	nders, gas l	lines)			
☐ Pressurized vessels or	systems					
☐ Vacuum chambers or s	ystems with >10	00 J stored	ener	ду		
☐ Autoclaves						
☐ Other (specify):						
Fire Hazards (check all that apply) ☑ None						
☐ Open flames ☐ Welding, Brazing, Silver Soldering						
☐ Flammable gases/liquids/solids ☐ Other spark producing activity						
☐ Other (specify):						
Chemical Hazards (check all that apply) ☑ None						
☐ Carcinogens	☐ Highly acute toxins			☐ Reproductive toxin	ıs [	☐ Corrosives
☐ Flammable liquids	☐ Flammable solids			☐ Strong oxidizers		□ Oils
☐ Explosives	☐ Peroxidizables			☐ Pyrophoric materia	als [	⊐ PCBs

☐ Asbestos	☐ Pesticides/herbicides		des	☐ Controlled substances		ed substances
☐ Highly reactive material	lighly reactive materials			□ Perchlorates		
☐ Storage or use of Beryllium or Beryllium articles. Attach Beryllium Use Review Form if			se Review Form if checked.			
☐ Toxic metals (e.g., As, E	Ba, Be, C	Cd, Cr, Hg, Pb	, Se, Ag)	)		
☐ Other (specify):						
Ionizing Radiation (check	all that	apply)		✓None		
☐ Sealed radioactive sour	ces		1		Windowles	s radioactive sources
☐ Dispersible radioactive	materials	3	1		Neutron-en	nitting radioactive sources
☐ Non-fissionable radioac	tive mate	erials	1		Fissionable	e radionuclides
☐ Ionizing radiation-gener	ating dev	vices (x-ray so	ources, ac	СС	elerators)	
☐ Other (specify):						
Nonionizing Radiation (c	heck all	that apply)			None	
☐ Class II, IIIa, or IIIb (visi	ble <15n	nW) lasers	1	☐ Class IIIb (nonvisible >15mW) or IV lasers		
☐ Dynamic magnetic fields >1G at 60 Hz or dynamic electric fields > 1kV/m at 60 Hz						
☐ Static magnetic fields < 5 G. No Exposure Form is required						
☐ Static magnetic fields > 5 G and < 600 G		!	☐ Static magnetic fields exposure. Attach Static Magnetic Fields Exposure Form when required.			
☑ Static magnetic fields ≥ 600 G						
☐ Radio frequency (RF) or Microwave sources exceeding 10 mW radiated output						
☐ Infrared sources > 10 W			☐ Ultraviolet sources > 1 W			
☐ Extremely low frequency (ELF) radio sources						
☐ Other (specify):						
Biological Hazards (check all that apply) ☑ None						
☐ Regulated etiological agent ☐ Recombinant DN		ant DNA	IA		☐ Animals	
☐ Human blood/components, human tissue/body fluids		y fluids	☐ Human subjects			
☐ Other (specify):						
Offsite Work (check appro	opriate b	ox)	✓ None	е		
☐ Reviewed or controlled at the offsite location	by ES&⊦	l programs	☐ Requ			al controls (include in the next
See <u>Identification of Signification of </u>			spects an	<u>าd</u>	Impacts Su	bject Area or your ECR if you

need assistance completing the following table.

Significant Environmental Aspects (check all that apply) ☐ None
-----------------------------------------------------------------

☐ Any amount of hazardous waste generation
☐ Any amount of radioactive waste generation
☐ Any amount of mixed waste generation (radioactive hazardous waste)
☐ Any amount of transuranic waste generation
☑ Any amount of industrial waste generation (e.g., oils, vacuum pump oil)
☐ Any amount of Regulated Medical Waste (including sharps, hypodermic needles or syringes)
☐ Any atmospheric discharges that require engineering controls to reduce hazardous air pollutants or radioactive emissions, or are identified as a Title V emission unit, or require monitoring under NESHAP
☐ Any liquid discharges that require engineering controls to limit the quantity or concentration of the pollutant, or include radionuclides detectable at the point of discharge from the facility, or contain any of the chemicals listed on BNL's SPDES permit
☐ Storage or use of any chemicals or radioactive materials that require engineering controls – see Storage and Transfer of Hazardous and Nonhazardous Materials Subject Area
☐ On-site or off-site transportation of chemicals or dispersible radioactive materials
☐ Any use of once-through cooling water with a flow of 4 gpm – 24 hrs/day (10 gpm – 8 hrs/day, daily use of >15 gpm for >60 days) and discharging to the sanitary sewer
□ Soil contamination or activation
☐ Any underground pipes/ductwork that contains chemical or radioactive material/contamination
□ Other environmental aspects related to your work (specify):
□ Process Assessment Form required (determined by ECR or other qualified person)

### III. DEVELOP AND IMPLEMENT HAZARD CONTROLS

For each hazard identified in the previous section, describe how that hazard is controlled. Identify the Engineering Controls (e.g., interlocks, shielding), Administrative Controls (e.g., procedures, RWPs) or Personal Protective Equipment (e.g., respirators, gloves; see the Personal Protective Equipment Subject Area) that will be employed to reduce hazards to acceptable levels.

The Experiment Review Coordinator, along with the **Principal Investigator** (PI) and Building Manager, as appropriate, will evaluate this experiment for impacts that will require an update to the Facility Use Agreement (FUA), and or Fire/Rescue Run Cards.

The **PI** develops and implements hazard controls in consultation with, and using feedback from, the personnel who will be performing the work.

A. Physical Hazards/Controls

Hazard	Controls (Administrative, Engineered, Protective Equipment)
Magnetic fields up to 70 kOe concentrated in small sample space. Fields around the machine is much smaller, several Oe – see Attachment 5	Environmetal magnetic shield (part of MPMS machine) knowing hazards and following operational procedures (Attachments 2 and 6).
Cryogens: Liquid Helium and Nitrogen	Knowing hazards and complying with safe operating procedures (Attachments 2, 4 and 6), protective equipment: gloves and goggles. Wearing long pants and shoes.

Note: Include maintenance, inspection and testing, and formal calibration, including frequency as appropriate.

#### B. Chemical Hazards/Controls

Hazard	Controls (Administrative, Engineered, Protective Equipment)
None	

Note: Refer to the Working with Chemicals Subject Area for requirements regarding particularly hazardous chemicals such as carcinogens, reproductive toxins, and highly acute toxins, including postings, decontamination plan, and address above.

### C. Environmental Hazards/Controls

Hazard	Controls (Administrative, Engineered, Protective Equipment)
None	

Note: Identify the requirements from applicable waste management subject area (<a href="https://next.org/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.com/nazardous.pic.c

### D. Radiation Hazards/Controls

Hazard	Controls (Administrative, Engineered, Protective Equipment)
None	

Note: List sources/materials. Attach or refer to Radiation Work Permits.

#### E. Biological Hazards/Controls

E. Diological Hazards	Controls
Hazard	Controls (Administrative, Engineered, Protective Equipment)
None	

Note: List additional approvals/permits/reviews required (e.g., BNL Biosafety Committee approval).

### F. Offsite Work Hazards/Controls

	11. 0.0
Hazard	Controls (Administrative, Engineered, Protective Equipment)
None	

<u>Note</u>: List the location of all off-site work and identify any off-site organization whose ESH requirements will be followed (e.g., other DOE Labs). Indicate additional controls (not specified above) that are needed.

### IV. PERFORM WORK WITHIN CONTROLS

All work shall be performed within the controls identified within this document. It is the PI's responsibility to ensure that this document is kept up to date. The PI should consult with the ERC as appropriate to determine if changes to this document are significant enough to require a new review/document.

If a hazard assessment may be required for this experiment, the PI should contact the ES&H Coordinator and/or the ERC for assistance. The PI should document any hazard assessments performed for this experiment in Section VI.

### A. Training

List all project personnel, indicating they are authorized and competent to perform the work described. List the training required for each individual. Identify any certifications or experiment-specific training required. Indicate if any project personnel are minors (under 18 yrs. of age). Contact your Training Coordinator and ES&H Coordinator as appropriate for assistance.

It is the responsibility of the PI to maintain a complete up-to-date list of personnel and their full training requirements, and to ensure that training and qualifications are maintained.

Name	Life/Guest #	Required Training (Course or JTA code)
Cedomir Petrovic	22904	TQ-COMPGAS1, HP-OSH-025, TQ-ODH, TQ-SMF
Al Langhorn	18811	TQ-COMPGAS1, HP-OSH-025, TQ-ODH, TQ-SMF
Tom Vogt	20358	TQ-COMPGAS1, HP-OSH-025, TQ-ODH, TQ-SMF
Yongjae Lee	22728	TQ-COMPGAS1, HP-OSH-025, TQ-ODH, TQ-SMF
Sangmoon Park	3714	TQ-COMPGAS1, HP-OSH-025, TQ-ODH, TQ-SMF
Markus Huecker	22589	TQ-COMPGAS1, HP-OSH-025, TQ-ODH, TQ-SMF
Kim Mohanty	15362	TQ-COMPGAS1, HP-OSH-025, TQ-ODH, TQ-SMF
Jessica Thomas	22954	TQ-COMPGAS1, HP-OSH-025, TQ-ODH, TQ-SMF
Genda Gu	22459	TQ-COMPGAS1, HP-OSH-025, TQ-ODH, TQ-SMF
Richie Rothe	17682	TQ-COMPGAS1, HP-OSH-025, TQ-ODH, TQ-SMF

<u>Note</u>: The <u>BNL Training and Qualifications Web Site</u> contains course offerings and descriptions, required training checklist, as well as employee training records.

### B. OSHA/DOE Required Medical Surveillance

Indicate if potential exposure is in excess of trigger levels listed. Exposure evaluation and/or medical surveillance may be required. Additional <u>training</u> may be required for any indicated agent. See the <u>SBMS</u> for additional information and controls on the hazards listed.

Regulated Hazard	Hazard Specific Training Trigger	Medical Surveillance Exposure Trigger
✓ None		
☐ Inorganic Arsenic	Any day above the OSHA action level (without regard to respirator use)	30 days/year above the action level (without regard to respirator use)
☐ Biohazards (CDC/NIH/WHO listed Agent)	None	See Subject Area for guidance
☐ Cadmium	Any day above the OSHA action level	30 or more days/year at or above the action level
Lasers	Use Class IIIb or Class IV Lasers	Use Class IIIb or Class IV Lasers
Lead	Any day above the OSHA action level	30 or more days/year at or above the action level
		- 30 days/year at or above the action level
☐ Methylene Chloride	Any day above the OSHA	<ul> <li>10 days/year above the 8-hour TWA PEL or the STEL</li> </ul>
a montylene emonde	action level	<ul> <li>Any time above the 8-hour TWA PEL or STEL for any period of time where an employee at risk from cardiac disease or other serious</li> </ul>

Regulated Hazard	Hazard Specific Training Trigger	Medical Surveillance Exposure Trigger
		MC-related health condition and employee requests inclusion in the program
□ Noise	Any day above the ACGIH TLV	Any time equal or greater then 85 dBA TWA 8-hour dose
☐ OSHA Regulated Chemicals  Acrylonitrile Benzene  Benzidine 1,3 Butadiene	Any day above the OSHA PEL	Routinely above the action level (or in the absence of an action level, the PEL)
4-Dimethyl aminoazobenzene Ethylene oxide Ethyleneimine Formaldehyde Vinyl Chloride	Any day above the ConA r LL	Event such as a spill, leak or explosion results in the likelihood of a hazardous exposure
		<ul> <li>Any time at ≥ 0.5 mT (5 G) for Medical Electronic Device wearer</li> </ul>
		- Any day at ≥ 60 mT (600 G) to whole body [8 hour average]
☐ Static Magnetic Fields	Worker who routinely works in magnetic field	- Any day at ≥ 600 mT (6000 G) to limbs [8 hour average]
		- Any Time at ≥ 2 T (20,000 G) to whole body [ceiling]
		- Any time at ≥ 5 T (50,000 G) to limbs [ceiling]

### C. Emergency Procedures

Identify any emergency actions, procedures, or equipment that must be in place to insure personnel safety and environmental protection. Include the Building Local Emergency Plan, location of emergency shutoffs, and spill control materials.

Appropriate work routines and safety precautions will be followed (see Attachments). Helum transfer will be performed with door wide open. Transfer dewar should be periodically inspected.

### D. Transportation

Identify materials, hazards and controls for any on-site and off-site transportation of hazardous and/or radioactive materials. See relevant SBMS Subject Areas.

There will be no transport of materials around the lab. However, cryogen storage dewars will be moved inside Physics building slowly and with care.

### E. Notifications

The PI or designee should notify building occupants of any activities that might impact them or their work, and document this here. List external personnel/organizations that require notification related to experimental activities and/or to be notified of changes (e.g., a BNL Committee for review/approval, Occupational Medicine Clinic, Fire/Rescue).

None.

### F. Termination/Decontamination

Describe any decommissioning plan, including decontamination of the area at termination of the experiment. Identify any hazards and controls, special precautions or procedures. Include chemical and waste reconciliation. Indicate if a walk-down or an ERE will be scheduled to ensure the area is suitable for future projects. Indicate if Work Permit Form/Procedure will be used.

No decontamination needed. Daily cleanup (wiping) will be done and areas will be thoroughly cleaned before release to other workers.

### G. Community Involvement Issues

Identify issues that may require community involvement (see the <u>Community Involvement in Laboratory Decision-making Subject Area</u>) and describe the plan that addresses these issues. Attach the Community Involvement Checklist.

None.

# V. PROVIDE FEEDBACK ON ADEQUACY OF CONTROLS AND CONTINUE TO IMPROVE SAFETY MANAGEMENT

Provide comments on the review process, including this form and communication. Identify any lessons learned or worker feedback contributing to modifications/improvements to the controls or process.

Formality of the review process prevents addressing real safety concerns. It could be significantly improved if safety committee would include members with considerable experience in experimental process which is being reviewed.

### VI. ATTACHMENTS

Use this section to include any supporting documents, hazard assessments, figures, tables, etc. that were not entered into the previous sections of the form.

# **<u>Attachment 1</u>**: Sample Signature Sheet.

The PI is responsible for communicating the requirements in this Experiment Safety Review (ESR) to the project workers. One way to accomplish this is to have each worker read the ESR and sign an agreement form, such as the one below. This sheet is **not submitted** as part of the review process.

I have read this Experimental Safety Review document and understand the hazards associated with my work activities and the controls in place to mitigate those hazards. I understand the environmental aspects of my work activities and will continually work to minimize waste generated and look for areas of improvement. I am aware of the training requirements and will maintain my qualifications.

Print Name	BNL#	Signature	Date

# <u>Attachment 2</u>: Safe Operating Procedures (Hazard Assessment)

The MPMS superconducting magnet produces extremely strong magnetic fields. Following safety procedures regarding the magnet is critical to laboratory safety. Furthermore, the MPMS utilizes cryogenic liquids for temperature control. There are several concerns surrounding cryogens: they burn skin on contact and they expand rapidly when warmed. Certain safety precautions are necessary when dealing with the liquid helium (and the liquid nitrogen, if a nitrogen-jacket dewar is included in the system) that the MPMS requires. Also, general electrical safety procedures should be followed, since the MPMS has several pieces of electronic equipment.

The most useful pieces of advice concerning safety are to use common sense and to be aware of the system's state and of your surroundings. If the system behavior appears abnormal, something may be wrong with the MPMS. If so, consider whether or not the problem poses a threat to personnel in the laboratory and take appropriate action. For the most part, the MPMS is provided with safety features to keep accidents from causing injury or serious equipment damage. Read the precautions below carefully and keep them in mind whenever working with the MPMS. Inexperienced users should be supervised.

### **Console Stability Safety**

The MPMS XL Control Console has been designed for ready access to boards and assemblies. The three front panels each tilt down to access the circuit board assemblies. In order to facilitate the removal of assemblies, the console has been designed with equipment mounted on drawers with slides. There is considerable weight in each drawer, and when more than one drawer is pulled forward, the console may tip forward, damaging personnel and equipment.

There is an internal latch preventing the drawer from sliding out. A tool is necessary to release the catch. A later section will discuss access to these components in more detail. There are CAUTION labels inside the drawer front panels, as well as inside the rear door to the console interior.

### **Cryogenic Safety**

The precautions for handling liquid helium are related to the very low temperatures of the liquid and its treatment as a high-pressure gas. Direct contact with surfaces or materials just removed from the helium bath, or exposure to high boil-off gas flows will freeze the skin almost instantly. Protection in the form of insulated gloves and safety eye glasses should always be employed.

Helium gas is an odorless, colorless, inert material that is non-flammable. While the gas itself is not toxic it can displace air in confined areas, potentially causing asphyxiation.

Transferring liquid helium from one container to another, or any operation that produces a high boil-off of gas from the liquid, should be performed in open shop or laboratory areas. Lack of oxygen causes dizziness, unconsciousness or death. More importantly, in an oxygen-depleted atmosphere, the body will not experience the buildup of carbon dioxide that normally produces respiratory distress. In this case a person may simply lose consciousness before realizing that anything is wrong. When using liquid helium, always use proper ventilation.

Because helium has a relatively low latent heat of vaporization, care must be exercised in the design and use of apparatus that contains it. Pressure-relief valves must be adequately sized to allow for sudden vaporization of liquid. Both the TCM and helium dewar provided by Quantum Design employ relief valves and burst disks to provide safe operation if there is either a leak into any of the insulating vacuum spaces or the superconducting magnet quenches. Any user-supplied equipment that utilizes an open volume exposed to liquid, particularly sealed volumes that might contain a small leak, should be similarly protected.

Liquid helium is the coldest liquid that will exist at atmospheric pressure and is therefore a very effective cryopump. A vessel containing liquid helium left open to the atmosphere will rapidly condense and solidify air and other gases. This can easily plug pressure relief passages, transfer ports, and other components of the gas handling system. Since helium gas is constantly evaporating from the liquid, high pressures within the container develop quickly. It is therefore essential to maintain a positive pressure (above atmospheric pressure) within any storage container and to make certain that all ports and orifices, except proper relief valves, remain closed when immediate access to the helium is not required.

Helium-cooled surfaces exposed to the atmosphere can similarly attract and condense air. Because nitrogen has a lower boiling point than oxygen, this gas will evaporate first leaving an oxygen-enriched residue that can flow onto surrounding surfaces. Contact with spontaneously combustible materials such as oil or grease can produce ignition, therefore possible exposed surfaces should be clean and free of such materials.

### **Magnet Safety**

Because the superconducting MPMS magnet can trap magnetic flux, it is possible to leave a charged magnet completely unconnected to the rest of the system. Doing so leaves no way to discharge the magnet directly, so avoid this practice. Never disconnect a charged magnet from the magnet controller and do not disconnect any of the other connections in the system while a magnet is charged. Several different cables contain connections for magnet control. Be sure to drive the magnet to zero field before disconnecting any cables if the probe ever needs to be disconnected from the controllers for any reason.

Superconducting magnet supplied with the MPMS is capable of disturbing computer monitors, affecting electron microscopes, erasing credit cards, attracting ferromagnetic tools, etc. **Transverse magnets produce substantially stronger fields surrounding the dewar than longitudinal magnets do.** Keep in mind that the magnet in your system produces strong fields that are not completely confined to the system unless it contains some type of magnetic shielding.

It is recommended that the magnetic field around the MPMS be measured and a line drawn to denote the region outside which the magnetic field does not **exceed** five gauss. The determination of where this line lies is the user's responsibility, since it varies from system to system. It is typically found about 1-2 m (3.3-6.6 ft.) from the edge of the dewar. **No heavy ferromagnetic objects (e.g. gas cylinders, large tools, etc.) should be brought within this region when the magnet is charged.** Gas cylinders in the laboratory should be secured to the walls and only informed personnel should be allowed to use large tools in the presence of the MPMS. It is possible to cause injury to personnel and damage to MPMS equipment by allowing heavy objects to be attracted to the MPMS.

Furthermore, the magnetic fields produced by the MPMS can be dangerous or fatal to wearers of pacemakers and other electrical or mechanical medical devices. **Anyone wearing a pacemaker or similar device should stay at least 3.0-4.5 meters (10-15 feet) away from the MPMS dewar.** This information should be posted in the laboratory where the MPMS is operated so that people wearing such devices are aware of the presence of large magnetic fields.

One effect to consider when first installing the system is that of the superconducting magnet's field on various equipment and objects in the laboratory. The following table shows the magnetic field required to produce certain effects and the radial distance from the dewar at which these fields were measured for a system with a nine tesla longitudinal magnet at full field. This list is far from comprehensive, so consider <u>all</u> of the equipment in your laboratory that might be affected by magnetic fields.

**Table 1-1** Possible Effects of the MPMS Magnet

Effect	Field Required (Gauss)	Approximate Distance from Dewar (cm)
Effects on electron microscope	1	216
Disturbance of color computer monitors	1-3	152
Disturbance of monochrome computer monitors	3-5	127
Erasure of credit cards, bank cards, etc.	10	102
Effects on watches and micromechanical devices	10	102
Lowest known field affecting a pacemaker	17	84
Erasure of magnetic tapes	20	79
Saturation of transformers and amplifiers	50	58
Erasure of floppy disks	350	30

### **Electric Safety**

The MPMS components are powered by standard 120 or 240 VAC power lines. These voltages are potentially lethal and appropriate care should be exercised around the equipment. Electronic equipment should be powered down and

unplugged before opening or removing any covers. Liquids should be kept away from the computer and electronics cabinet. Frayed or damaged cords should be replaced immediately.

# **Attachment 3: System Description**

The Quantum Design Magnetic Property Measurement System (MPMS) is a sophisticated analytical instrument configured specifically to study the magnetic properties of small experimental samples over a broad range of temperatures and magnetic fields. The system has two major hardware components: (1) the MPMS dewar and probe assembly, and (2) the associated control system in the MPMS control console. These components are shown in Figures in Attachment 3: System description. Automatic control and data collection are provided by an computer and two independent subsystem controllers, the Model 1802 and Model 1822. Gas control and other ancillary functions in the system are also automated.

Within the dewar, a cryogenic probe integrates a superconducting magnet with a SQUID detection system and a high-performance temperature control system. The system provides rapid precision measurements over a temperature range of 1.9 to 400 Kelvin (1.9 to 350 Kelvin for MPMS<sub>2</sub>). Modular design of the system also allows the probe to be easily refit for additional options, or disassembled for repair.

### **Description**

When installed, two cabinets plus the computer comprise the complete MPMS system, occupying an area approximately 1.5 meters by 3 meters. The system has five major physical components as follows:

- 1. The computer with the installed MPMS control system software. This unit communicates with the two MPMS subsystem controllers via the IEEE-488 Bus Interface protocol, typically referred to as the General Purpose Interface Bus (GPIB).
- 2. The electronic control console is comprised of the Model 1822 MPMS controller, the Model 1802R/G bridge, the MPMS gas control system and vacuum pump, the superconducting magnet power supply, and the microstepping sample transport controller.
- 3. The liquid helium dewar mounted in its cabinet.
- 4. The MPMS cryogenic probe which includes the Temperature Control Module (TCM) integrated with the MPMS superconducting magnet and SQUID detection system.

5. The sample transport mechanism which mounts on the top of the TCM, and three sample rod assemblies for mounting samples.

The cables connecting the MPMS probe and sample transport to the control cabinet are color-coded for convenience. Note that the two gas lines are different sizes and should be connected with the ninety degree elbow at the control console.

The computer should be connected to the system printer and to the MPMS console with the two GPIB cables provided. The GPIB cable connecting the Model 1802 and Model 1822 controllers is installed at the Quantum Design factory.

While the components may be easily assembled during installation, the installation will normally be performed by Quantum Design service representatives. This also provides the opportunity for user training and an opportunity to discuss some of the measurement techniques we have developed.

When configured for magnetic susceptibility measurements, the MPMS integrates seven major functional systems under the MPMS control software. These are as follows:

- 1. The Temperature Control Module (TCM) which provides an actively regulated, precision thermal environment over its entire range of operation, 1.9 to 400 K (1.9 to 350 K for MPMS<sub>2</sub>)
- 2. The superconducting magnet system which provides reversible field operation up to plus and minus 7 tesla (depending upon system configuration) using an oscillatory technique to minimize magnet drift immediately following field changes.
- 3. The SQUID detector system which includes the Model 2000 SQUID Amplifier control electronics, sensing pick-up loops, and specially designed filtering with full computer control via the interface computer.
- 4. The sample handling system (sample translator and sample transport) which allows automatic sample measurements and position calibrations using a microstepping controller having a positioning resolution of .0003 cm.
- 5. The gas handling system which provides gas flow control for temperature regulation, flushing, and cleaning procedures.
- 6. Liquid helium system which provides refrigeration for the superconducting detection system and magnet, as well as providing for operation down to 1.9 Kelvin.
- 7. The computer control system which includes the computer with a hard disk, a 3.5 inch microfloppy drive, a printer, and the integrated MPMS control system operating software.

The MPMS system; comprising the control console, dewar cabinet, and computer; requires a lab space about 1.5 meters by 3 meters. It should be positioned two to three feet from any wall to allow reasonable access to the rear of the unit. In addition, the dewar cabinet should be conveniently accessible from the front and one side to facilitate sample insertion/removal and transferring of liquid helium. Ready access to the system for liquid helium storage

dewars is essential since the MPMS dewar must be refilled with helium on a regular basis, approximately every 5 to 7 days (depending on use). The MPMS dewar has a capacity of 56 liters, with helium consumption ranging from about 4.5 liters/day when operating the system at low temperatures and up to 6 to 7 liters/day when operating at room temperature. The dewar is shock-mounted in its enclosure, so that no special vibration isolation is required in normal laboratory environments.

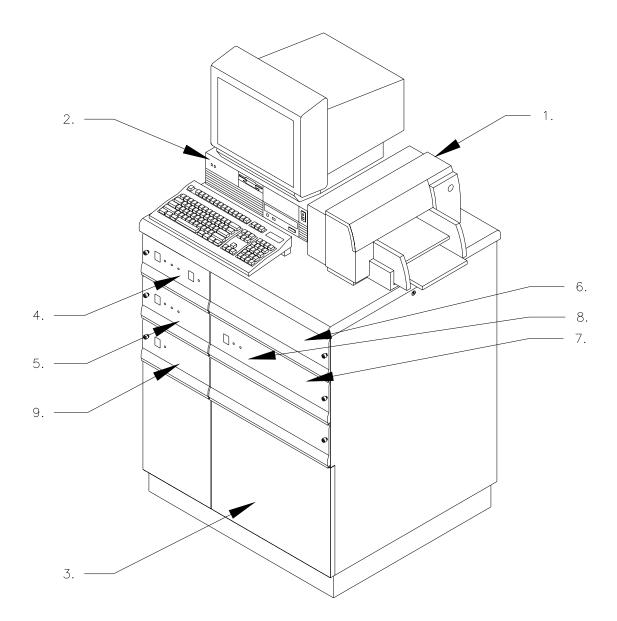
The computer can either sit on top of the control console or on a nearby desk. The two meter GPIB cable supplied with the system can be replaced with a cable up to four meters long if desired. The MPMS control console is connected to the probe in the dewar cabinet by two flexible metal vacuum lines bundled with several multi-conductor electrical cables. After the cables and vacuum lines are installed, the two cabinets should be separated as far apart as possible without straining either the electrical cables or vacuum lines. Since the control console contains a mechanical pump and other ferrous materials, the separation prevents magnetic signals (especially from the vacuum pump) from interfering with the SQUID detection system.

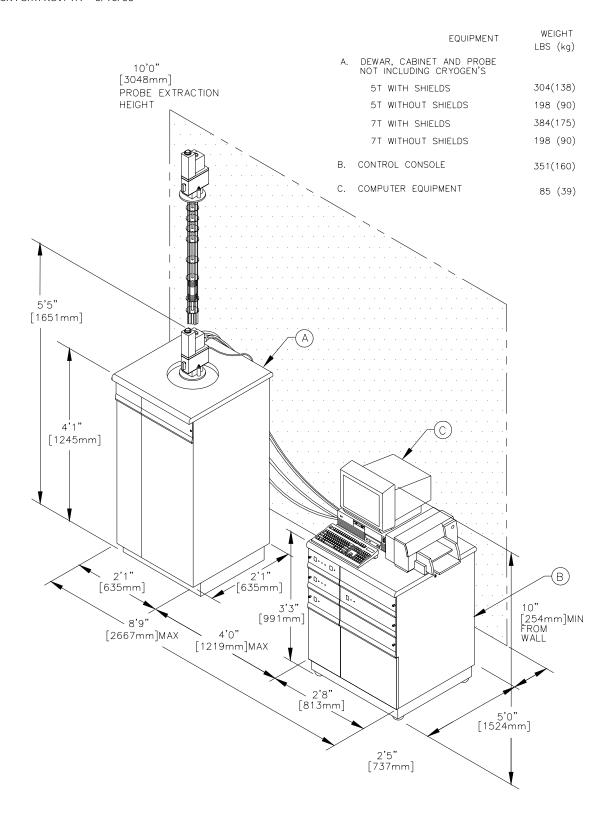
A three meter (ten feet) ceiling height is also required to allow sample insertion and liquid helium transfers. The sample rod, which is a rigid tube slightly less than 1.2 meters in length, must be inserted into the top of the MPMS probe. A three meter ceiling provides the clearance needed to avoid bending the sample rod.

During operation the MPMS power requirement is 15 amps at 110 VAC, single-phase. (The unit can also be configured for 220 volt service at 7.5 amps.) The control console is supplied with a single eight-foot power cord that plugs into a receptacle at the rear of the control console; three additional AC receptacles are required for the computer, its monitor, and the printer. Under normal operation, the unit requires no external gas lines, but a dewar of helium gas with a low-pressure regulator (0 to 10 psi) and a modest length of flexible plastic hose is needed when cooling the unit down. The small Alcatel vacuum pump, located inside the control console, is fitted with a replaceable exhaust filter and should not require venting to the outside atmosphere. A fitting is provided for attachment to a helium recovery system if desired. No water service is required.

The MPMS has been designed to operate without a superconducting magnetic shield around the SQUID detection coils, allowing magnetic field changes and sample measurements to be executed in rapid succession. Rejection of interference from nearby magnetic sources is achieved by using a small spacing and high degree of balance in the second-derivative detection coils. Nonetheless, large magnetic sources (such as elevators or automobiles) can interfere with measurements if they are located very near the instrument. To avoid this type of interference, the instrument should be located at least several meters from any such large magnetic objects. Smaller items such as steel frame chairs or steel lab stools should be kept several feet away, or, at least, kept stationary while sample measurements are being performed.

- Printer
   Comput
   Vacuum
- Printer
   Computer
   Vacuum Pump
   Model 1822 Controller
   Model 1802 R/G Bridge
   Gas/Magnet Control
   Magnet Power Supply
   Power Distribution Unit
   RSO Controller

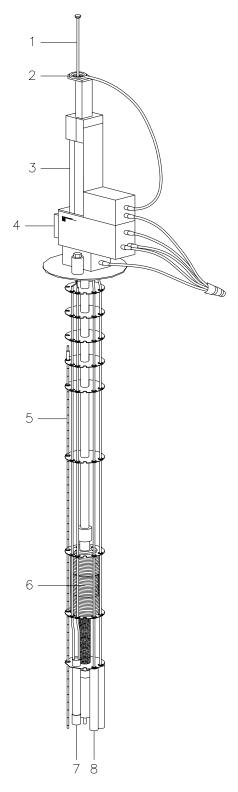


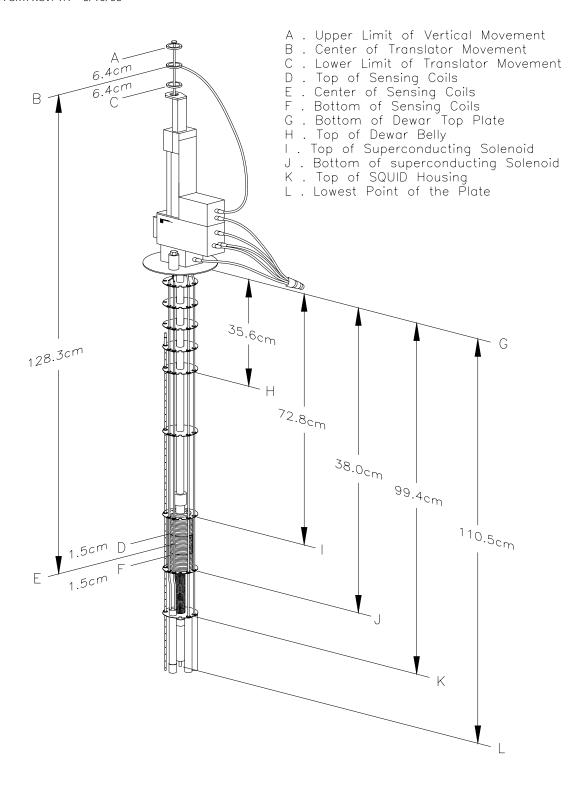


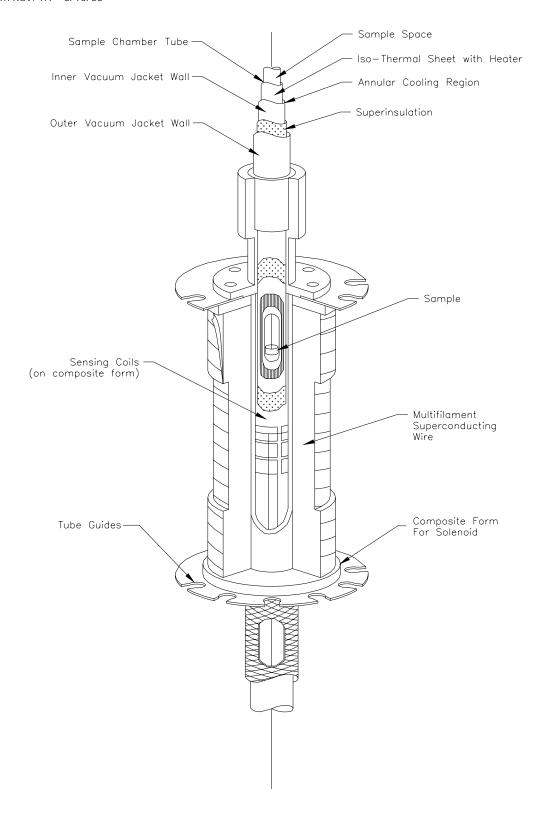
### SYSTEM COMPONENTS

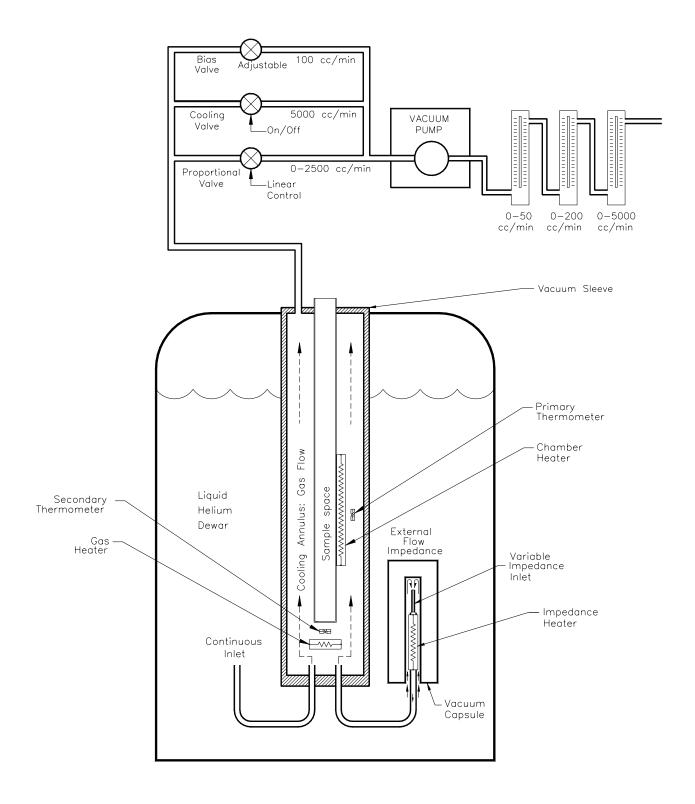
- Sample Rod
   Sample Rotator
   Sample Transport
   Probe

- 4. Probe5. Helium Level Sensor6. Superconducting Solenoid7. Flow impedance8. SQUID Capsule









# **<u>Attachment 4</u>**: Helium Transfer Procedures

### The Initial Transfer

The initial transfer will require between 75 and 90 liters of liquid helium to cool down the system and fill the dewar.

For the first transfer, the output end of the transfer tube should be fitted with one of the extensions provided to reach to within a few inches of the bottom of the MPMS dewar (about 44 inches (111.76 cm) below the top surface of the dewar). Just before the transfer tube is inserted into the dewar, the plug should be removed from the MPMS top plate, and the transfer fitting installed. It should seat in the tube sticking up from the top plate, and the horizontal tube should be directed away from the head. If a helium recovery system is being used, you may wish to connect it to this exhaust fitting. Open the cooling valve from the **Diagnostic Gas Controls** menu. First press **F7** for the **Diagnostic Menus**, move cursor to **Gas Controls** and press **Enter**. Next move the cursor down to the **Cooling Valve** selection and press **Enter**. Select **Open** and press **Enter** again. This will provide full helium gas flow and help prevent blocking the impedance tube.

Insert one end of the transfer tube into the storage dewar and the other end into the transfer port. Insert the transfer tube into the MPMS dewar slowly, making sure that its extension does not bump into the magnet or other parts attached to the lower part of the probe. The transfer should be started slowly to take maximum advantage of the cold helium gas. Since the initial transfer will take 2 to 3 hours, it is helpful to have a small fan or a hair dryer directed across the head during the transfer to help keep excessive moisture from condensing on the head of the probe.

The transfer rate can be set by adjusting the pressure in the storage dewar. While the dewar and probe are cooling down, the pressure should be held at about 200 mm of mercury. This should produce a steady gas flow out of the transfer fitting. When liquid begins to collect, normally after about 1 hour, the flow will momentarily drop to zero, then resume. Also, if the helium level is being plotted on the computer, it will begin to increase at this time. As the liquid helium level increases, the gas flow out of the pump will increase to a maximum of 2.5 liters/min. Once this flow rate has been achieved and the helium level is greater than 25%, the cooling valve can be closed. Close the cooling valve from the **Diagnostic Gas Controls** menu. First press **F7** for the **Diagnostic Menus**, move cursor to **Gas Controls** and press **Enter**. Next move the cursor down to the **Cooling Valve** selection and press **Enter**. Select **Close** and press **Enter** again. The SQUID should also be tuned at this time as described in the MPMS Software User's Manual Chapter 9. Continue the transfer until the helium level reading reaches 100% or until the level stops increasing indicating an empty storage dewar.

It will take 24 to 48 hours for the dewar to completely cool down and equilibrate, and a high but decreasing boiloff should be expected during this period. The quiescent boiloff of the

system with the sample region cold (less than about 200 Kelvin), is about five liters of liquid per day. This loss is higher when the sample tube is warm or when the system is being cooled down in the high-power cooling mode.

### **Subsequent Transfers**

The helium level in the system should be kept at about 40% or higher to provide cooling for the superconducting magnet. If the level drops below 40%, the magnet should not be used. Subsequent helium transfers into a cold MPMS dewar are essentially the same as the initial transfer with the following exceptions:

- 1. The magnetic field should be set to zero before beginning the transfer. If the magnet is left at a high field when the transfer is started, the initial process of inserting the warm transfer tube into the dewar may cause the magnet to quench.
- 2. A small wire or toothpick should be slipped into the dewar relief valve to release the dewar pressure before removing the transfer port plug.
- 3. The output end of the transfer tube only needs to get into the belly of the MPMS dewar (about 16 inches (40.64 cm) below the top plate), so no extension should be used in subsequent transfers.
- 4. Before the transfer tube is inserted into the storage dewar, it should be cooled by blowing cold gas through it until a plume appears at the outlet. If the warm tube is inserted first, the initial warm gas from the tube will boil off liquid already in the MPMS dewar.

At the beginning of the transfer, before inserting the transfer tube into the storage dewar, remove the screw cap, compression ring, and o-ring from the top of the transfer fitting, and slip them up over the output end of the transfer tube. With the transfer fitting in place, after the tube has been precooled, the tube can be slipped into the transfer fitting, and the screw cap tightened to seal the o-ring around the transfer tube.

<u>Note</u>: If you try to insert the transfer tube through the o-ring while cold gas is coming out, the o-ring will freeze to the transfer tube, causing it to jam as it is going through the fitting. Be sure the o-ring, the compression ring, and the screw cap are installed over a warm transfer tube.

After the transfer tube has been inserted into the MPMS dewar, the pressure in the storage dewar can be set to about 1 psi. The progress of the transfer can be monitored on the computer with the helium level plot. At the transfer rate produced by the 1 psi storage dewar pressure, it will take 30 to 40 minutes to fill the dewar when starting at a level of 40 percent.

Note: If the exhaust of the transfer tube is connected to a helium recovery system, a pressure greater than 1 psi may be required to transfer helium. Higher pressure may be needed to overcome backpressure from the helium recovery system; typically only 2 or 3 psi would be required.

# <u>Attachment 5</u>: Stray fields around Quantum Design Measurement Systems

For comparison, this table includes fields around other Quantum Design machines which use similar magnet systems. It is important to notice that system used in this experimental safety review (MPMS 7T) has environmental magnetic shield. Thus, stray field distances are expected to be half of the ones in the table.

Magnet Size	1 Gauss, Radial	5 Gauss, Radial	1 Gauss, Axial	5 Gauss, Axial
MPMS 7T	45 ln(114.3	20 ln. (50.8	20.5 ln.(52	2 In. (5.1 cm)
	cm)	cm)	cm)	
PPMS 7T	94 ln(238.8	40 ln(101.6	59 ln(149.9	25 In. (63.5
	cm)	cm)	cm)	cm)
PPMS 9T	94 In(238.8	40 ln(101.6	63 In (160 cm)	30 In. (76.2
	cm)	cm)		cm)
PPMS 14T	118 ln	70.9 ln(180	91.8	55.1 ln(140
	(300cm)	cm)	In(233cm)	cm)
14T Shielded	70.9 In	33.5 In (85cm)	49.2 In	31.5 ln (80
	(180cm)		(125cm)	cm)
14T,.1%;5.5c	120 In(305cm)	66.5ln (169	120ln (305	68 In (173 cm)
m		cm)	cm)	
(AMI)				
14T,.1%;5.5c	93 In (236 cm)	41.5ln (105	75 In	43 In (109 cm)
m		cm)	(190.5cm)	
Shielded				
(AMI)				

# **<u>Attachment 6</u>**: Operating Procedures

### **Mounting Samples**

Samples may be mounted on the end of the sample rod using a variety of techniques. Because of the wide variations in the types, sizes, and geometries of samples from customer to customer, we do not supply a standard sample holder. However, there are several techniques that previous customers have found useful and these are provided as a guide for your own adaptation.

One particularly useful sample holder is a thin-walled quartz tube. This can be attached by mounting a non-magnetic coupling on the end of the sample rod to mate the quantalloy portion of the rod and quartz tube together. The sample can then be held in place by inserting it into the quartz tube and stuffing quartz wool

in beneath it. A similar sample mounting can be accomplished with something as simple as a soda straw.

Another technique that can be used with tiny samples is to attach them to the side of a small quartz rod with a dot of vacuum grease. If this is done, centering rings should be attached to the top and bottom of the rod to ensure that the sample does not get scrapped off the rod as it is inserted into the sample chamber.

If either of these techniques is used, the sample should be mounted about 7 to 10 cm (2.8 to 3.9 inches) from the end of the sample holder so that the end of the sample holder does not move through the detection coils during the measurement. The end of the sample holder will also produce a signal in the SQUID detector. If it approaches the coils closely enough to be detected, the signal will be distorted and the magnetic moment calculation for the sample will be in error.

Larger samples can be suspended from the end of the sample tube with white cotton thread. However, this allows the sample to swing back and forth in the sample chamber, causing corresponding oscillations in the SQUID detection system. This can cause measurement errors much larger than when using rigidly mounted samples. Generally, we have found it much more desirable to use a rigid mounting rather than suspending samples from a thread.

For additional information about sample mounting please refer to Quantum Design Application Note 1014-201 "MPMS Sample Mounting Considerations."

### **Taking A Measurement**

Once the sample is mounted on the sample rod, the following procedure describes the procedure for making a measurement:

### Installing the Sample Rod Assembly into the Sample Chamber

- 1. Tighten the knurled nut on the slide seal assembly so that this portion of the sample rod is positioned approximately 122 cm (48 inches) away from the center of your sample.
- 2. Pull the sample rod up through the slide seal so that the sample holder is drawn into the protective glass sleeve (the shroud). Make sure the top of the sample holder is flush against the bottom of the blue plug.
- 3. Vent the sample chamber.
  - a. If there is a sample present in the sample chamber, proceed to step 4.
  - b. If there is no sample present in the sample chamber and the READY LED is on, vent the system by closing the airlock valve (this causes the sample chamber to vent automatically). Close the airlock by rotating the handle counter clockwise so that it points (horizontally) to the indicated CLOSED position. Wait for the VENTING LED to turn off.

- c. If there is no sample present in the sample chamber, the airlock is already in the CLOSED position, and the green READY LED is on; toggle the handle to OPEN then back to CLOSED. Wait for the VENTING LED to turn off.
- 4. Open the slide seal clamps on the sample transport socket block so the blue plug or slide seal assembly may be released.
- 5. Remove the blue plug or sample rod.
- 6. Before proceeding, verify that the three o-rings in the blue plug socket block are in place, free of debris, and not dry. If necessary, apply a small amount of Apiezon M grease to the surface of the o-rings.
- 7. Insert the sample rod into the sample chamber until the slide seal housing can be secured into the socket of the sample transport.

Note: Ensure that the white "dot" (of the blue plug) is facing forward. This ensures proper gas flow through the slide seal. It may be secured by rotating the two rectangular handles on the socket block so that the slide seal is forced down against the three o-rings in the block by cam action.

- 8. The blue plug of the slide seal assembly should be flush to the top of the socket block.
- 9. Close the slide seal clamps on the sample transport so the slide seal assembly may be fastened.
- 10. Press the button on the front of the MPMS probe labeled PURGE AIRLOCK to initiate automatic purging of the airlock. When the green LED labeled READY comes on, open the airlock valve by rotating the handle clockwise so that it points vertically to the indicated OPEN position.

Note: If the READY LED does not light within a few seconds after the purging sequence ends, there may be a leak in the sample handling system or the annulus pressure gauge may be negative. Negative pressure is characteristic of acquiring low temperatures. The temperature must be stable before the READY LED will light.

11. Lower the sample slowly and slowly turn the rod during the insertion process.

**Note:** If the rod appears to be dry or is not moving smoothly through the lip seals, place some Apiezon M grease above the blue plug.

12. Lock the clip assembly into the actuator shoe by tightening the thumb nuts.

### **Center the Sample**

- 1. Access the **Register Sample** menu (**F2**) and perform the following actions:
  - a. Turn **Autosample Tracking** on. If it is already on, toggle it off then on again to ensure it is recognized by the MPMS software.
  - b. **Initialize Sample Transport** in order to calibrate the transport to 0.27 cm from the bottom limit of the transport movement range. This position is high enough above the transport limit to allow for movement to correct for thermal contraction in the sample rod when the temperature is lowered.
- 2. Perform the center sample process; it may be necessary to apply a magnetic field. Briefly, the sample centering process includes performing a full length DC scan, adjusting the sample position, and then performing DC centering scan. (See the MPMS software manual for more information.)

### **Take Measurements**

Collect data by opening the **Measure** menu (**F3**), then moving the cursor to **Immediate** and press **ENTER**. Data may also be collected through a sequence.

Retrieving Samples And Cleaning The Sample Chamber

If a sample falls off the sample holder or the sample chamber becomes contaminated, the MPMS is designed to allow the sample chamber to be opened to room temperature without danger of plugging small gas tubes or otherwise degrading the operation of the system. Simply set the system temperature to room temperature or slightly higher (about 310 Kelvin); when the system becomes stable at that temperature, the airlock can be opened to the room.

Note that when the airlock is vented after closing the airlock valve, the airlock flushing valve is left closed, which is its energized state. For this reason, when there is no sample in the unit we recommend that the airlock plug (provided with the system) be installed in place of the slide seal assembly, followed by purging the airlock. When the system is left in this condition, open the airlock valve slightly, then close it again to vent the airlock. The airlock can now be opened to the room. The airlock flushing and venting valves can also be controlled from the **Diagnostic Gas Controls** menu under **Flush Valve** and **Vent Valve**, respectively (access the **Diagnostic Menus (F7)**, move cursor to **Gas Controls**, and then press **Enter**).

Once the airlock is open and the temperature is set to 310 Kelvin, a rod with an appropriate hook or spiral wire can be inserted to the bottom of the sample chamber to retrieve lost samples, or a Kimwipe with a mild solvent can be used to swab out the sample chamber. If a solvent is needed, use it sparingly since the o-ring seals at the top of the sample chamber around the airlock valve may be damaged by strong solvents.

After the lost sample has been retrieved or the sample chamber cleaned out, replace the airlock plug in the top of the sample transport mechanism, and with the airlock open press the **PURGE AIRLOCK** button. This will purge the entire sample chamber of air and any vapors remaining from the solvent, and it will restore the static helium pressure of a few millimeters. You may wish to repeat this process a few times, allowing time for any residual liquid or water moisture to vaporize. When this process is finished, close the airlock valve.



Building 911B P.O. Box 5000 Upton, NY 11973-5000 Phone 631 344-4250 Fax 631 344-5954 lessard@bnl.gov

Managed by Brookhaven Science Associates for the U.S. Department of Energy

Date:

August 14, 2003

To:

T. Sheridan, Deputy Director for Operations

From:

July Awbland, Actor, Environment, Safety and Health Committee

Subject:

LESHC 03-04, Recommendation for Approval of Operation for the Physics

Department Magnetic Properties Measurement System

The Cryogenic Safety Subcommittee of the BNL ES&H Committee has reviewed the installation of a Physics Department Magnetic Properties Measurement System (MPMS) in our meeting of July 28, 2003. The MPMS is a commercially available unit that is designed to measure the magnetic properties of small samples. The unit has completed the required commissioning activities and is awaiting approval for operation.

The Meeting Minutes are attached for your information.

The Committee recommends granting approval for MPMS operations, subject to the following conditions.

The Physics Department Principal Investigator will:

- Maintain conformance to the Static Magnetic Field Subject Area, the ODH Subject Area and personnel training requirements, as appropriate for MPMS operation. -Complete<sup>1</sup> except for the ODH requirement<sup>2</sup>.
- 2. Maintain MPMS relief valve piping, ductwork, or deflectors to redirect the discharge of cryogens away from personnel Complete<sup>1</sup>.
- 3. Provide the liquid helium dewar relief valve orifice area calculation, based on the maximum credible energy deposition rate in the helium volume **Complete**<sup>1</sup>.
- 4. Reformat Attachments 2-6 of the MPMS Experimental Safety Review Form into a procedure. (The ESR is included as Appendix 2 to these Minutes.) Complete<sup>1</sup>
- 5. Submit a Piping and Instrument Drawing (P&ID) to the Committee Complete<sup>1</sup>.

CC w/ attachment (via Email):

<sup>1</sup> This Condition has been completed in the interim between our July 28<sup>th</sup> meeting and the date of this letter.

providing adequate air flow/exchange in the vicinity of the MPMS, or

The ODH calculations must be revised based on the test results of the selected ventilation configuration.

<sup>The Committee requires:
Confirmatory testing of the temporary ventilation configuration to assure that the high velocity fan is</sup> 

Completion and confirmatory testing of the permanent ventilation supply and exhaust for the MPMS room

### LESHC Members

- S. Aronson
- M. Beckman
- L. Hinchliffe (BAO)
- M. Hucker
- T. Kirk
- L. Marascia
- T. Monahan
- S. Musolino
- C Petrovic
- S. Shapiro
- T. Sheridan
- J. Tarpinian
- M. Zarcone



Building 460 P.O. Box 5000 Upton, NY 11973-5000 Phone 631 344-8627 Fax 631 344-2361 sheridan@bnl.gov

managed by Brookhaven Science Associates for the U.S. Department of Energy

# Memo

date:

August 18, 2003

to:

Sam Aronson

from:

Thomas R. Sheridah

subject:

Approval of Operation for the Physics Department Magnetic Properties

Measurement System (MPMS)

reference:

Memo; E. Lessard (LESHC) to T. Sheridan, dated 8/14/03

After review of the recommendation of the Cryogenic Safety Subcommittee of the Laboratory Environment, Safety & Health Committee (LESHC) (referenced memo above), I authorize approval of operation for the MPMS, and understand that LESHC conditions 1-5 have been completed, with the exception of the ODH requirement noted in condition 1 as follows:

### The Committee requires:

- Confirmatory testing of the temporary ventilation configuration to assure that the high velocity fan is providing adequate air flow/exchange in the vicinity of the MPMS, or
- Completion and confirmatory testing of the permanent ventilation supply and exhaust for the MPMS room.

The ODH calculations must be revised based on the test results of the selected ventilation configuration.

### TRS/lim

cc:

T. Kirk

E. Lessard

R. Travis

### NOTES:

- 1. LHE CAPACITY = 44 LITERS.
- 2. LHE THEORETICAL STATIC BOIL-OFF = 1.80 L/DAY.
- 3. LN2 CAPACITY = 33.9 LITERS.
- 4. LN2 THEORETICAL STATIC BOIL-OFF = 1.69 L/DAY.

fiberglass walls to include a diffusion barrier (.0005" 300 series grade stainless steel or equivalent.)

6 MASK ALL THREADED HOLES PRIOR TO PAINTING.

 $\stackrel{\wedge}{7}$  FINISH: PAINT PER QD-SPEC-1001-09.

8. BAKE-OUT AND DIFFUSION PUMP FOR 24 HOURS.

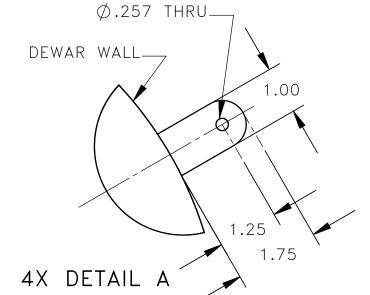
BELLOWS SEALED EVACUATION VALVE MUST BE INSTALLED PARALLEL TO TOP SURFACE OF DOME AS SHOWN.

APPLY TEFLON TAPE TO PIPE THREADS PRIOR TO INSTALLING CAP AND RELIEF VALVES.

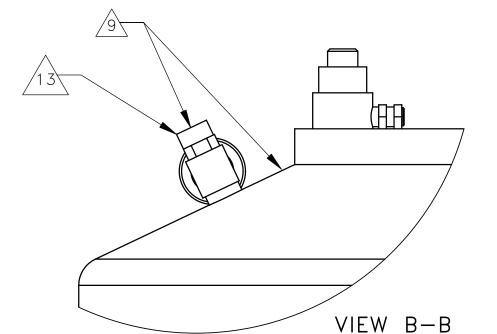
11 SERIAL NUMBER TO BE ENGRAVED INTO DEWAR NECK.

12. DEWAR TO BE SHIPPED UNDER VACCUM.

MANUFACTURER TO APPLY A GENEROUS AMOUNT OF GREEN TORQUE SEAL (ANTI—SABOTAGE LACQUER) TO THE BELLOWS SEALED EVACUATION VALVE.



	REVISIONS							
LTR	DESCRIPTION	DATE	APPROVED					
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D	SEE ECO 6442							



32/	32/	
		16/

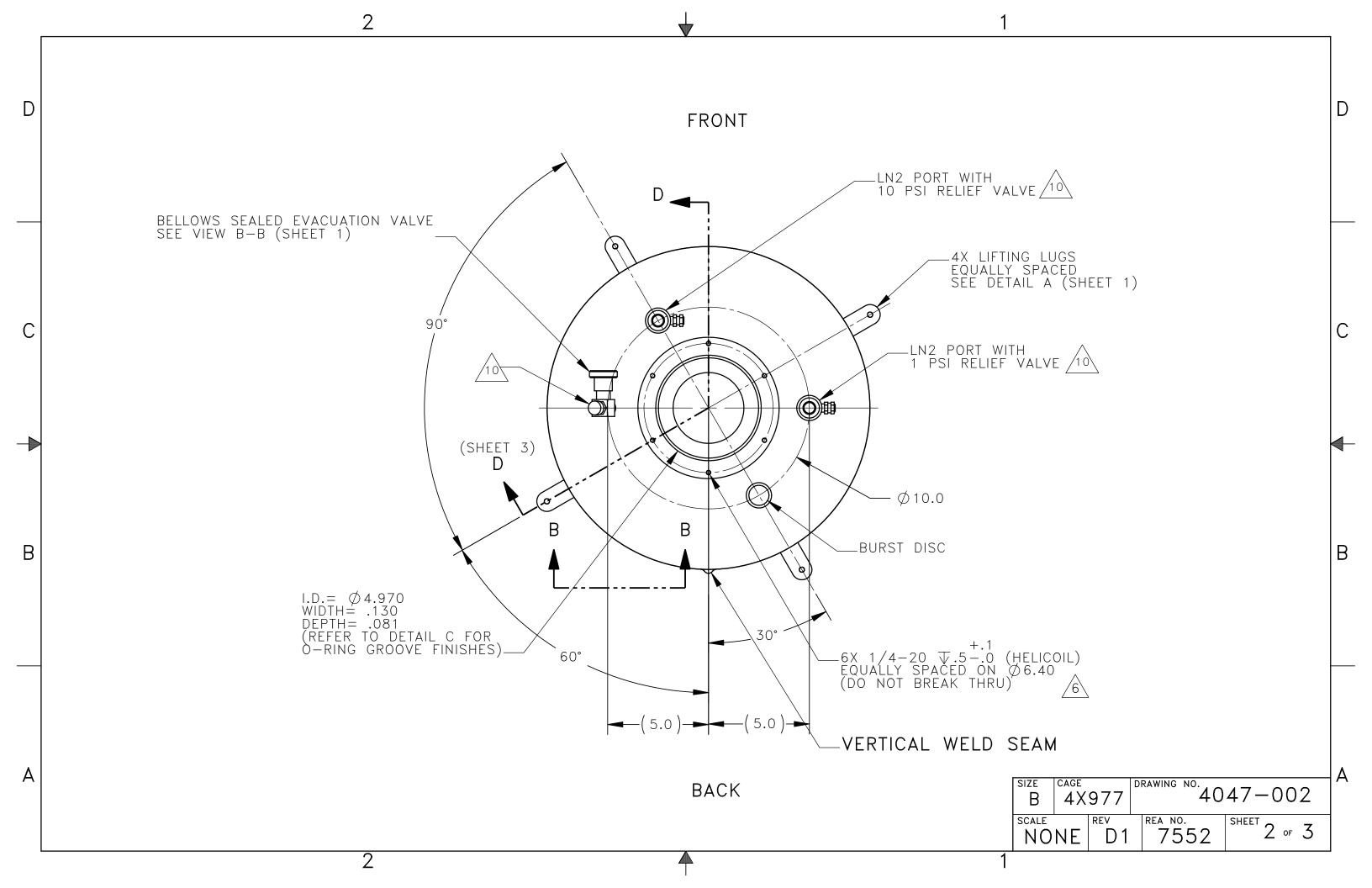
DETAIL C
(O-RING GROOVE FINISHES)

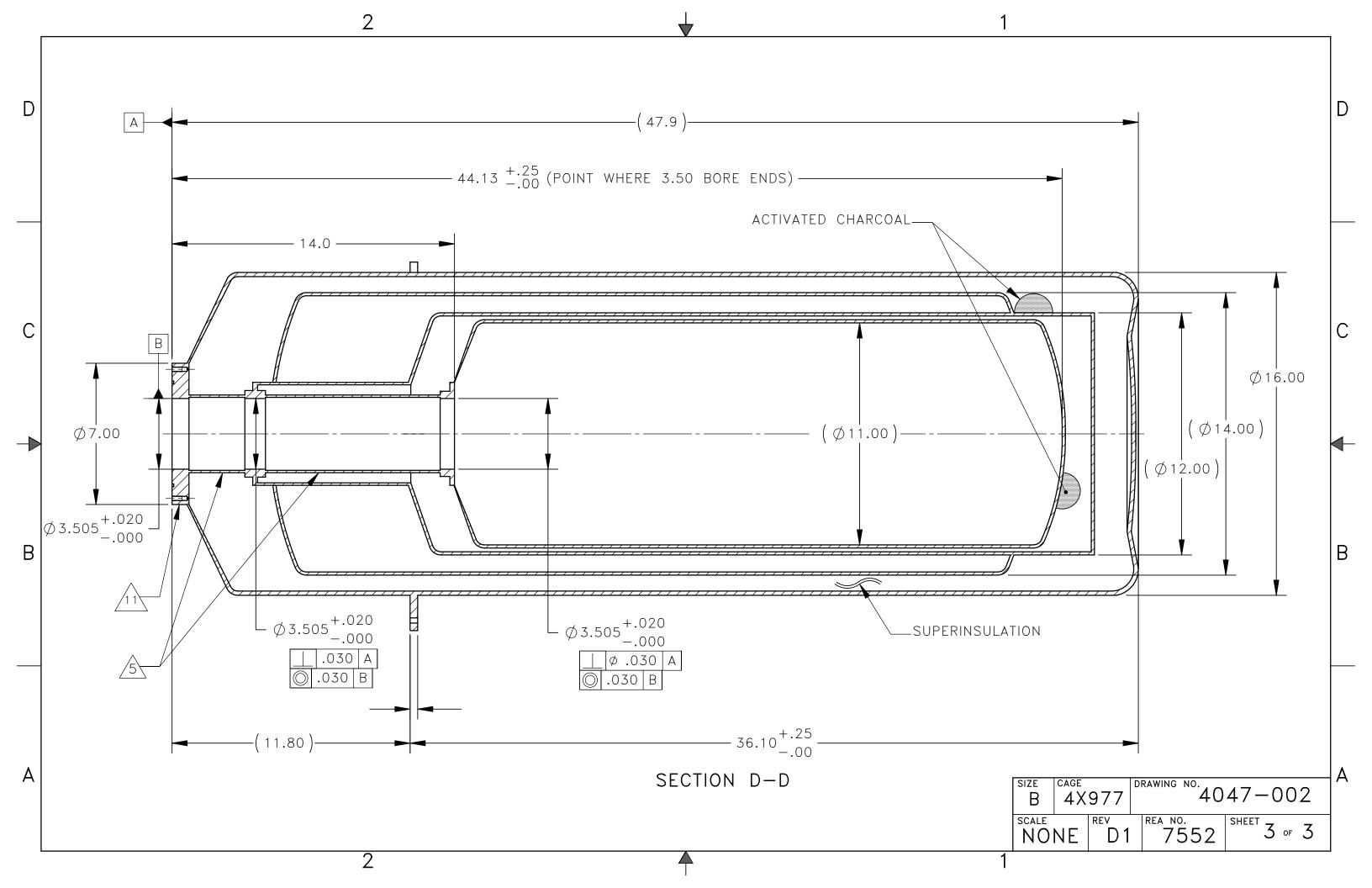
APPROVED SOURCE OF SUPPLY					
CONTROL	VENDOR	VENDOR	ITEM		
P/N	ADDRESS	CAGE CODE	PART NO.	IDENT.	
4047-002	KADEL ENGINEERING CORP 1627 EAST MAIN STREET DANVILLE, INDIANA 46122 TEL: (317) 745-2798 FAX: (317) 745-2799	N/A	KC190	4047002 4X977	

# SOURCE CONTROL DRAWING

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63/				B		977	40	47-002	
DO NOT SCALE DRAWING	─ END	ITEM	MPMS	SCALE		REV	REA NO.	SHEET	
PREPARED IN SOLIDWORKS 200	1 NEXT	ASSY L	JSED ON	NO	NE	D1	7552	1 of 3	

2





Tel: 858.481.4400 800.289.6996 Fax: 858.481.7410 E-Mail: info@qdusa.com http://www.qdusa.com/



August 6, 2003

Cedomir Petrovic Department of Physics, Bldg. 510B Brookhaven National Laboratory Upton, NY 11973

Hello Cedomir,

I am providing you some pressure relieve valve calculations in the hope that it will help fulfill all the safety requirements needed for your usage of the MPMS Quantum Design system.

As explained before there is a two-stage pressure relieve valve (part number 4014-026), which is used for the evaporation of helium gas. The first stage is a 0.25in. diameter opening while the second stage has a diameter of 0.625 in. diameter. The first stage has a rating of 1500 liters/min while the second has a rating of 5000 liters/min. There is a maximum of 2 liters of liquid Helium evaporation on a full magnet quench. Assuming a 1 to 700 relation of liquid to gas of Helium there would be a total of 1400 liters evaporating within in a span of 2-3 minutes from the dewar. Clearly the first stage valve (rating at 1500 L/min) could handle the quench but as a safety design we have establish a bigger air flow pressure valve of 5000 L/min which will handle the release in the event that it is necessary. From Quantum Design extensive service and product life there has never been an incident where safety design have become a concern, since the proper precautions are take from the very first design stage. That is from the dewar design and the pressure release mechanism to auto-shut off command within the software. If further detail information is necessary please talk to our engineer, Jost Diederichs at extension 125.

Sincerely,

Socrates Gomez Sales Engineer